PROGRESS IN NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

An International Review Journal

VOLUME 29

SUBJECT INDEX, AUTHOR INDEX
CONTENTS OF PREVIOUS VOLUMES

Executive Editors
J. W. EMSLEY and J. FEENEY

Consulting Editor
L. H. SUTCLIFFE

ELSEVIER

Copyright © 1996 Elsevier Science B.V. All rights reserved. 0079-6565/96/$32.00 PER ARTICLE

This journal and the individual contributions contained in it are protected by the copyright of Elsevier Science B.V., and the following terms and conditions apply to their use:

Photocopying
Single photocopies of single articles may be made for personal use as allowed by national copyright laws. Permission of the Publisher and payment of a fee is required for all other photocopying, including multiple or systematic copying, copying for advertising or promotional purposes, resale, and all forms of document delivery. Special rates are available for educational institutions that wish to make photocopies for non-profit educational classroom use.
In the USA, users may clear permissions and make payment through the Copyright Clearance Center, Inc., 222 Rosewood Drive, Danvers, MA 01923, USA. In the UK, users may clear permissions and make payment through the Copyright Licensing Agency Rapid Clearance Service (CLARCS), 90 Tottenham Court Road, London W1P 0LP, UK. In other countries where a local copyright clearance centre exists, please contact it for information on required permissions and payments.

Derivative Works
Subscribers may reproduce tables of contents or prepare lists of articles including abstracts for internal circulation within their institutions. Permission of the Publisher is required for resale or distribution outside the institution.
Permission of the Publisher is required for all other derivative works, including compilations and translations.

Electronic Storage
Permission of the Publisher is required to store electronically any material contained in this journal, including any article or part of an article. Contact the Publisher at the address indicated.
Except as outlined above, no part of this publication may be reproduced, stored in a retrieval system or transmitted in any form or by any means, electronic, mechanical, photocopying, recording or otherwise, without prior written permission of the Publisher.

Disclaimers
No responsibility is assumed by the Publisher for any injury and/or damage to persons or property as a matter of products liability, negligence or otherwise, or from any use or operation of any methods, products, instructions or ideas contained in the material herein.
Although all advertising material is expected to conform to ethical (medical) standards, inclusion in this publication does not constitute a guarantee or endorsement of the quality or value of such product or of the claims made of it by its manufacturer.

⊙ The paper used in this publication meets the requirements of ANSI/NISO Z39.48—1992 (Permanence of Paper).

Printed in The Netherlands

ELSEVIER

PROGRESS IN NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

Subject Index, Volume 29

Ab initio calculations. *See* Chemical shift calculations
Absolute shielding scales 251–3
Acid strength
  Lewis acid-Lewis base interactions 163–4
  medium effects 160–1
  reaction rates 156–7
Acidity 129–68
  electrochemical measurements 132–3
  solid surfaces 161–3
Acidity functions 130–2
  $\Delta\delta^{\circ}$ parameter 135–55
  Hammett acids 132–5, 155–6
  hydronation equilibria 157–60
  limitations 155–63
  NMR determination 133–5
  non-Hammett acids 155–6
  relative acid strengths 156–7
  superacid strength evaluation 157–60
Acute leukemia, bone marrow NMR 208–10
Additivity 262–4
Adenine H2 resonances 78–9
Amino resonances, RNA 87–8
Anomeric resonances 79
Anti-inflammatory drugs 21–9
Anti-viral compound BW524W91 31–2
Antipyrine 20–1
Arginine 45

Bacteriophage RNA polymerases 56–8
Base pair opening 103
Base resonances 66–8, 77–80
Base-to-base correlations 80
Base-to-sugar correlations 80
Biofluids 18–19, 19–33
Blood formation 171–3
Bone marrow
  composition 171–3
  function 171–3
  transplantation 210–13
Bone marrow NMR 169–227
  acute leukemia 208–10
  bone marrow transplantation 210–13
  bone tumors 215
  chemical shift selective imaging 197–8, 205–6
  diagnostic uses 206–21
  field mapping methods 198–204
  gradient-echo sequences 195–6
  ^{1}H spectroscopy 173–81
  magnetization transfer measurements 190–2, 204, 205–6
  metastases of malignant tumors 214–15
  osteochrondritis 215–16
  osteomyelitis 213–14
  osteonecrosis 214
  pathological diseases 208–14, 214–16
  relaxometric measurements 181–90
  SENEX method 197–8
  spin-echo sequences 194–5, 196
  trabecular structures 216–21
  volume selective ^{1}H spectroscopy 173–81
Bone tumors 215
Bony structures 173
Boron trifluoride hydrates 144, 149–50
Boron trifluoride-phosphoric acid-water mixture 144, 150–2
BW524W91, HPLC-NMR 31–2

Capillary electrophoresis (CE-NMR) 46–8
^{13}Carbon 129–68
  See also $\Delta\delta^{\circ}$ parameter determination of acidity functions
  carbon-based sugar assignments 68
  chemical shifts 135–55
  ^{1}H couplings 73, 74–5
  ^{31}P couplings 75–6

Catalysts 154
CE. *See* Capillary electrophoresis
Chemical shifts 229–78
  See also $\Delta\delta^{\circ}$ parameter determination of acidity functions
  absolute shielding scales 251–3
  additivity 262–4
  applications 248–57
  categorization of contributions 257–9
  CSGT 242–3
  density functional theory 247–8
  future directions 268–74
  GIAO 238–40
  GIAO-MBPT(2) 245–6
  Hartree-Fock approaches 237–43
  4-hexen-3-one 140, 141
  IGLO 240–1
  intermolecular effects 249–51
  intramolecular effects 248–9
  large system calculations 257–68
  local origin methods 237–8
  locally dense basis set approach 259–60
  LORG 241–2
  MC-IGLO 246–7
  mesityl oxide 139, 141
  molecular dynamics methods 271–3
  new experiments 273–4
  proteins 264–8, 268–70
  scaling 262–4
  secondary isotope effects 255–7
  selective imaging 197–8, 205–6
  shielding
    absolute scales 251–3
    hyperpolarizabilities 260–2
    polarizabilities 260–2
    properties 233–5
    sensitive probe function 235–7
    surfaces 253–5
  SOLO 243–5
  temperature dependence 255–7
Chemical synthesis, RNA 55–8
Chromatographic separations 1–49
  capillary electrophoresis 46–8
  future developments 47–8
  high performance liquid chromatography 8–41, 47–8
  solid phase extraction chromatography 3, 4–6
  supercritical fluid chromatography 4, 41–5
  supercritical fluid extraction 4, 6–8
Complete relaxation matrix analysis 94–6
Continuous set of gauge transformations (CSGT) method 242–3
CSGT. *See* Continuous set of gauge transformations

Density functional theory 247–8
Di-allyl phthalate, SFC-NMR 43
Dihedral angle constraints, RNA structure determination 97–102
Dimer forms 90
Drug glucuronides 39–41
Drug metabolites 32–3, 34
Dynamic HPLC-NMR 39–41
$\Delta\delta^{\circ}$ parameter determination of acidity functions 135–55
  boron trifluoride hydrates 144, 149–50
  boron trifluoride-phosphoric acid-water mixture 144, 150–2
  calibration 140–3
  development 135–40
  fluoroboric acid 147–8
  hydrogen bonding ability 154–5
  indicator concentration 143–6
  industrial catalysts 154
  methanesulfonic acid 140–3
  methanesulfonic acid-phosphorus pentoxide system 152–4
  methanesulfonic acid-sulfolane mixtures 154
  perchloric acid 140–3, 144
  phosphoric acid 144, 146–7
  phosphotungstic acid 148–9
  principle 135–40
  second hydronation of unsaturated ketones 146
  sulfuric acid 140–3, 144
  testing 140–3

Eaton Catalyst 152
Electrochemical acidity measurements 132–3
Enzymatic ligation 61–2
Enzymatic synthesis 55–8

Field mapping methods 198–204
^{19}Fluorine NMR, HPLC-NMR 22, 23, 24
Fluoroboric acid 147–8
Flurbiprofen 21–9

Gauge including atomic orbital (GIAO) method 238–40
Gauge including atomic orbital-second-order many-body perturbation theory (GIAO-MBPT(2)) 245–6
GIAO. *See* Gauge including atomic orbital
GIAO-MBPT(2). *See* Gauge including atomic orbital-second-order many-body perturbation theory
Gradient-echo sequences 195–6

Hammett acids 132–5, 155–6
Hartree-Fock approaches 237–43
Heteronuclear coupling constants 71–6
4-hexen-3-one, chemical shift differences 140, 141
High performance liquid chromatography (HPLC-NMR) 8–41, 47–8
  anti-inflammatory drugs 21–9
  anti-viral compound BW524W91 31–2
  antipyrine 20–1
  drug glucuronides 39–41
  early studies 9–10
  endogenous substances in biofluids 18–19
  flurbiprofen 21–9
  ^{1}H NMR 17, 24, 25, 26, 28
  ^{2}H NMR 32–3, 34
  ibuprofen 21–9
  in vitro metabolism studies 33, 34
  natural products analysis 33–6
  operational methods 11–15
  other chemical systems 38–9
  paracetamol 28, 29–31
  peptide libraries 35, 36–8
  polymers 38
  probe design 10–11
  schematic representation 7
  sensitivity 11, 15–18
  solvent problems 11
  xenobiotic substances in biofluids 19–33
High resolution NMR spectroscopy 2
Homonuclear coupling constants 71–6
HPLC-NMR. *See* High performance liquid chromatography
^{1}Hydrogen
  bone marrow 173–81
  ^{13}C couplings 73, 74–5
  HPLC 17, 24, 25, 26, 28
  ^{1}H couplings 72–4
  ^{31}P couplings 75–6
  SFC 42, 43, 45
  SPEC 3
^{2}Hydrogen, HPLC 32–3, 34
Hydrogen bonding 96–7, 154–5
Hydronation equilibria 157–60

Ibuprofen, HPLC-NMR 21–9
IGLO. *See* Individualized gauge for localized orbitals
Imino resonances, RNA 86–7
in vitro metabolism studies 33, 34
Individualized gauge for localized orbitals (IGLO) method 240–1
Industrial catalysts 154
Intermolecular effects 249–51
Interproton distances 90–6
Intrabase correlations 63–4
Intramolecular effects 248–9
Isotopic labelling
  enzymatic ligation 61–2
  NTP preparation 60–1
  RNA 58–62, 89–90
  rNTP synthesis 61
  spectral assignment 89–90

Ketones 146

Leukemia, bone marrow NMR 208–10
Lewis acid-Lewis base interactions 163–4
Ligands, RNA recognition 121–2
Local origin methods 237–8
Localized orbital local origin (LORG) method 241–2
Locally dense basis set approach 259–60
LORG. *See* Localized orbital local origin

Magnetization transfer measurements 190–2, 204, 205–6
MC-IGLO. *See* Multiconfiguration IGLO
Mesityl oxide 139, 141
Metastases of malignant tumors 214–15
Methanesulfonic acid 140–3
Methanesulfonic acid-phosphorus pentoxide system 152–4
Methanesulfonic acid-sulfolane mixtures 154
Molecular dynamics methods 271–3

Monomer forms, RNA 90
MT. *See* Magnetization transfer measurements
Multiconfiguration IGLO (MC-IGLO) 246–7

Natural products, HPLC-NMR 33–6
NOESY, RNA 54
Non-Hammett acids 155–6
NTPs. *See* Nucleotide triphosphates
Nucleotide triphosphates (NTPs) 60–1

Off-line chromatography-NMR analysis 4–8
Off-line NMR spectroscopy 2
On-line NMR spectroscopy 2
Osteochrondritis 215–16
Osteomyelitis 213–14
Osteonecrosis 214

Paracetamol 28, 29–31
Pathological diseases 208–14, 214–16
Peptides 35, 36–8
Perchloric acid 140–3, 144
Phosphoramidite chemistry 58
Phosphoric acid 144, 146–7
Phosphoric acid-boron trifluoride-water mixture 150–2
^{31}Phosphorus
  ^{13}C couplings 75–6
  ^{1}H couplings 75–6
Phosphorus pentoxide-methanesulfonic acid system 152–4
Phosphotungstic acid 148–9
Polymers, HPLC-NMR 38
Proteins
  chemical shifts 264–8, 268–70
  RNA-protein recognition 121–2
Proton exchange, RNA 103
Pyrimidine H5 and H6 resonances 79

Reaction rates, acidity functions 156–7
Relaxometric measurements 181–90
  human bone marrow 185–7
  longitudinal relaxation time 181–4
  short repetition times 187–90
  transverse relaxation time 184–5
Ribo-NTPs 61
RNA 51–127
  amino resonances 87–8
  backbone correlations 68–71
  bacteriophage T7 RNA polymerases 56–8
  base pair opening 103
  base resonances 66–8, 77–80
  base-to-base correlations 80
  base-to-sugar correlations 80
  ^{13}C-^{31}P couplings 75–6
  ^{13}C-based sugar assignments 68
  converged structures 112–14
  dihedral angle constraints 97–102
  dimer forms 90
  distance geometry 106–7
  dynamics 119
  enzymatic vs. chemical synthesis 55–8
  exchangeable proton resonances 86–9
  future prospects 119–22
  heteronuclear coupling constants 71–6
  homonuclear coupling constants 71–6
  hydrogen bonding constraints 96–7
  ^{1}H-^{13}C couplings 73, 74–5
  ^{1}H-^{1}H couplings 72–4
  ^{1}H-^{31}P couplings 75–6
  imino resonances 86–7
  interproton distances 90–6
  intrabase correlations 63–4
  isotopic labelling 58–62, 89–90
  monomer forms 90
  nitrogen resonances 86–9
  NTPs 60–1
  phosphoramidite chemistry 58
  protein recognition 121–2
  proton exchange 103
  purification 55–63
  restrained molecular dynamics 107–10
  rNTP synthesis 61
  sequential resonance assignments 82–6
  size limitation 120–1
  solvent suppression methods 64–6
  spectral assignments 76–90
  spin systems 80–2
  structure calculations 104–19
    converged structures 112–14
    distance geometry 106–7
    quality 110–12, 114–19
    restrained molecular dynamics 107–10
  structure determination 90–103
    additional constraints 102
    base pair opening 103
    dihedral angle 97–102

hydrogen bonding 96–7
interproton distances 90–6
proton exchange 103
structure quality 110–12, 114–19
sugar resonances 66–8, 77–80
sugar-to-sugar correlations 80–2
synthesis 55–63

Scaling 262–4
Second-order LORG (SOLO) 243–5
Secondary isotope effects 255–7
Semi-quantitative distance constraints, RNA
interproton distances 96
SENEX method 197–8
Sequential resonance assignments
backbone-directed 83–6
interproton distances 82–3
SFC. *See* Supercritical fluid chromatography
SFE-NMR. *See* Supercritical fluid extraction-NMR
Shielding
absolute scales 251–3
hyperpolarizabilities 260–2
polarizabilities 260–2
properties 233–5
sensitive probe function 235–7
surfaces 253–5
Solid phase extraction chromatography (SPEC-NMR) 3, 4–6
Solid surfaces, acidity 161–3
SOLO. *See* Second-order LORG
Solution structures, refinement 268
Solvent suppression methods 64–6
SPEC. *See* Solid phase extraction chromatography
Spectral assignment 63–71
backbone correlations 68–70
base resonances 66–8, 77–80
^{13}C-based sugar assignments 68
exchangeable protons 86–9
intrabase correlations 63–4
isotopic labelling 89–90
nitrogen resonances 86–9
sequential resonance assignments 82–6
solvent suppression methods 64–6
spin systems 80–2
sugar resonances 66–8, 77–80
Spin-echo sequences 194–5, 196
Sugar resonances 66–8, 77–80
Sugar-to-sugar correlations 80–2
Sulfolane-methanesulfonic acid mixtures 154
Sulfuric acid 131, 140–3, 144
Superacid strength 157–60
Supercritical fluid chromatography (SFC-NMR) 4, 41–5, 47–8
Supercritical fluid extraction (SFE-NMR) 4, 6–8

Temperature dependence 255–7
Trabecular structures 216–21
Tumors, bone marrow NMR 214–15

Volume selective ^{1}H spectroscopy
one pulse train methods 174–5
sequence parameters 180–1
spatially selective phase inversion 173–4
spectra from different sites 175–9

X-ray solution structures 268
Xenobiotic substances in biofluids, HPLC-NMR 19–33

ELSEVIER

PROGRESS IN NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

Author Index, Volume 29

Aboul-ela, F., 51
Allain, F.H.-T., 51

de Dios, A.C., 229

Fărcaşiu, D., 129

Ghenciu, A., 129

Lindon, J.C., 1

Nicholson, J.K., 1

Schick, F., 169

Varani, G., 51

Wilson, I.D., 1

ELSEVIER

PROGRESS IN NUCLEAR
MAGNETIC RESONANCE
SPECTROSCOPY

Contents of Previous Volumes

Volume 26

NMR of Noble Gases Dissolved in Isotropic and Anisotropic Liquids
J. JOKISAARI

Characterization of Biomolecular Structure and Dynamics by NMR Cross Relaxation
R. BRÜSCHWEILER and D. A. CASE

Artifacts in Two-dimensional NMR
A. D. BAIN, I. W. BURTON and W. F. REYNOLDS

Copper–Zinc Superoxide Dismutase: A Paramagnetic Protein that Provides a Unique Frame for the NMR Investigation
I. BERTINI, C. LUCHINAT and M. PICCIOLI

Variable Angle Sample Spinning NMR in Liquid Crystals
J. COURTIEU, J. P. BAYLE and B. M. FUNG

Multinuclear and Multidimensional NMR Methodology for Studying Individual Water Molecules Bound to Peptides and Proteins in Solution: Principles and Applications
I. P. GEROTHANASSIS

^{17}O NMR Studies of Hemoproteins and Synthetic Model Compounds in the Solution and Solid States
I. P. GEROTHANASSIS

Fluorine NMR of Proteins
J. T. GERIG

Isotope Labeling in Solution Protein Assignment and Structural Analysis
D. M. LEMASTER

Magnetically-oriented Phospholipid Micelles as a Tool for the Study of Membrane-associated Molecules
C. R. SANDERS II, B. J. HARE, K. P. HOWARD and J. H. PRESTEGARD

NMR Studies of Complex Surfactant Systems
O. SÖDERMAN and P. STILBS

NMR Studies of Translational Diffusion in Lyotropic Liquid Crystals and Lipid Membranes
G. LINDBLOM and G. ORÄDD

Recent Developments in Transferred NOE Methods
FENG NI

Volume 27

Selenium-77 Nuclear Magnetic Resonance Spectroscopy
H. DUDDECK

Chemical Shifts in Proteins Come of Age
L. SZILÁGYI

^{1}H NMR in the Structural and Conformational Analysis of Oligosaccharides and Glycoconjugates
E. F. HOUNSELL

^{1}H NMR Spectroscopy of Human Blood Plasma
M. Ala-Korpela
^{13}C Nuclear Magnetic Relaxation and Local Dynamics of Synthetic Polymers in Dilute Solution and in the Bulk State
P. Dais and A. Spyros
Hydration Water Molecules seen by NMR and by X-ray Crystallography
M. Billeter
^{2}H Zero Field NMR Spectroscopy
P. Jonsen

Volume 28
Milestones in the First Fifty Years of NMR
J. W. Emsley and J. Feeney
A Historical Account of NMR in the Solid State
E. R. Andrew and E. Szczesniak
The Development of Experimental and Analytical High Resolution NMR
J. N. Shoolery
A History of Biological Applications of NMR Spectroscopy
J. S. Cohen, J. W. Jaroszewski, O. Kaplan, J. Ruiz-Cabello and S. W. Collier
From NMR Diffraction and Zeugmatography to Modern Imaging and Beyond
F. W. Wehrli
Spin Dynamics of Exchanging Quadrupolar Nuclei in Locally Anisotropic Systems
B. Halle
Metabolite Profiling by One- and Two-dimensional NMR Analysis of Complex Mixtures
T. W.-M. Fan
NMR Studies of High T_c Superconductors
K. Asayama, Y. Kitaoka, G.-Q. Zheng and K. Ishida
Fluorine-19 Magic-Angle Spinning NMR
J. M. Miller
Lanthanide Induced Shifts and Relaxation Rate Enhancements
J. A. Peters, J. Huskens and D. J. Raber